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Clearinghouse for Federal Scientific and Technical Information
National Bureau of Standards, U. S. Department of Commerce
Springfield, Virginia 22151
Price: Printed Copy $3.00; Microfiche $0.65

ANL-7658
Reactor Technology

ARGONNE NATIONAL LABORATORY
9700 South Cass Avenue
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P. Vilinskas, E. C. Filewicz,*
and J. R. Humphreys**

Chemical Engineering Division

June 1970

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**Now with Division of Reactor Development and Technology, USAEC, Washington.

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SELECTED DATA AND DISCUSSION OF THE PLUGGING INDICATOR FOR SODIUM LOOPS

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ABSTRACT

The differences between manual, continuous, and oscillating plugging indicators in a sodium system are discussed briefly. The experimental data presented were obtained with the manual and the oscillating plugging indicators.

The initial results showed that temperatures of manual plugging indicators did not agree with cold-trap temperatures as required by theory. Manual plugging runs were made to determine reasons for the large variation in temperature difference. The cooling rates were varied while maintaining constant plugging-indicator flow rate and cold-trap temperature. These results indicated the trend that at low cooling rates (approaching zero) the plugging temperature would approach cold-trap temperature.

Work at Los Alamos with the oscillating plugging indicator indicated the reasons for the large variation observed with the manual plugging indicators. The manual plugging indicator used in our tests was modified into an oscillating plugging indicator, and successful runs were made in the temperature range of 269-400°F. Temperature changes of the cold trap, and hence the impurity concentration of the sodium in the system, were indicated on the temperature trace of the oscillating plugging indicator. The temperature differential between the cold trap and the manual plugging indicator was indeed reduced by operating in the oscillating mode.

Premature precipitation was identified as an important factor in the operation of the oscillating plugging indicator. If the heating and cooling area of the plugging indicator is such as to allow precipitation of the impurities upstream from the orifice, then it becomes impossible to operate the device in the oscillating mode. Also, species other than oxide were evident in the test runs.

I. INTRODUCTION

The plugging indicator is an instrument that measures the saturation temperature of an impurity in a liquid metal such as sodium. It consists of a constriction in a tube through which the metal flows, a means of cooling the metal and measuring its temperature at the constriction, and a flowmeter in series with the constriction to measure flow variations. As the liquid metal is cooled, impurity saturation temperatures are reached, and impurities precipitate and deposit in the constriction, thus reducing metal flow. The temperature at which the flow begins to decrease is called the "plugging temperature." For liquid sodium, the plugging temperature has been associated with the saturation temperature of sodium oxide, and oxide contents have been obtained from the solubility-temperature relationship.

As operating experience with manual models of plugging indicators in sodium increased, it became apparent that the plugging temperature was related not only to the oxygen concentration but also to the flow rate and cooling rate of the sodium. The plugging temperature, then, was not necessarily an accurate measure of an impurity concentration or of the true saturation temperature. The difference between the plugging temperature and the saturation temperature was clearly illustrated by work at Los Alamos[1] with an oscillating plugging indicator and by work in England[2] with the continuous plugging indicator. The manual plugging temperature was shown to be below the true saturation temperature and related to the finite concentration difference necessary to initiate precipitation in the orifice.

Deposits other than sodium oxide are possible in the plugging-indicator orifice and, sometimes, two different species may deposit at the same time. Usually, however, Na_2O is assumed to be the impurity precipitating, and the plugging temperature is converted into oxygen concentration units by means of oxygen solubility data, such as those of Rutkauskas.[3] In most instances, Na_2O precipitation may be a valid assumption, but more work is necessary to clarify this point. At present, the real value of the plugging indicator is not in determining oxide concentration, but in determining whether deposits are accumulating and thus causing sodium lines to plug. Also, the use of the plugging indicator in conjunction with the cold trap is very helpful. Both units operate on the same principle, and the plugging indicator is a convenient means of determining when the operation of a cold trap is advisable and when its effectiveness is minimal. As shown in Section III, when the plugging indicator is operated in conjunction with a cold trap, the saturation temperature of the plugging indicator usually corresponds to the cold-trap temperature.

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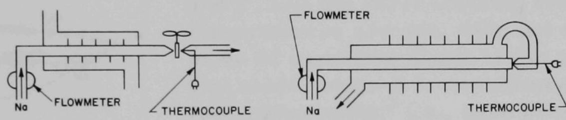
II. THE PLUGGING INDICATOR CIRCUIT AND MODES OF OPERATION

Figure 1 shows two models of a plugging indicator. The first, shown in Fig. 1A, consists of a flowmeter, a forced-air cooling section, an orifice valve, and a thermocouple. An orifice valve is a sodium service valve that has a notch ground into the stem so that when the valve is in the closed position the notch acts as an orifice. To clear the orifice, all one has to do is open the valve restoring the sodium flow. The second model, shown in Fig. 1B, incorporates a heat exchanger with cooling fins and an orifice plate with either a single opening or several openings. Both of these designs are representative of the many manual plugging indicators in service today.



A. Simple Plugging Indicator   B. Regenerative Plugging Indicator

Fig. 1. Component Arrangement in Typical Manual Plugging Indicators

As the sodium flows through a plugging-indicator circuit, it is cooled at one point to nearly its freezing temperature. The sodium entering the circuit is usually at 600°F. Thus, the maximum change in viscosity and density of the sodium as a result of cooling is from 0.284 to 0.705 cP and from 0.882 to 0.927 g/cm^3. The mass flow of sodium at any one point in the circuit will always equal the mass flow at any other point in the same circuit. The volume flow rate will change according to the density variations. Therefore, if a flowmeter that measures the volume flow rate is employed, it should be located in a constant-temperature region. If it is not, the maximum change in flow rate attributable to the change in sodium density in the flowmeter would be approximately 5%. In a single-orifice plugging indicator, where the largest pressure drop is across the orifice, the temperature-associated flow changes will be proportional to the square root of the change in sodium density. The maximum flow change due to this factor, assuming the change in sodium temperature is from 600 to 220°F, is 22%, as calculated by the orifice-flow equation.[4]

A. Manual Plugging Indicator

The manual plugging indicator is a plugging indicator of no particular design but of a particular mode of operation. The cooling is initiated manually, e.g., by activating a cooling fan, and subsequently the temperature at the orifice and the flow rate through the orifice are monitored until

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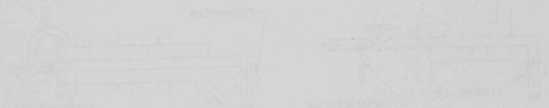

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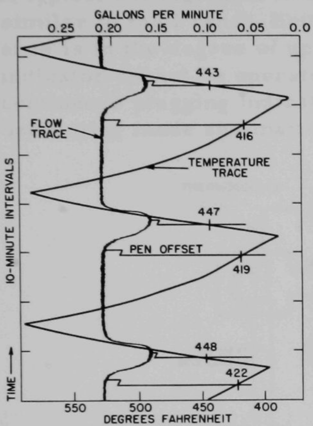

Fig. 2

Comparison of the Manual-plugging-indicator Temperature to the True Impurity-saturation Temperature as Measured by the Unplugging Mode

Figure 2 illustrates this phenomenon.* The conventional break temperatures for the three plugging runs shown are 422, 419, and 416°F, and the corresponding true saturation temperatures are 448, 447, and 443°F. The mode of operation shown is the partial oscillation mode, or as it is sometimes called, the unplugging mode;[2] i.e., once precipitation in the orifice has started, cooling is discontinued and heat is applied to redissolve the precipitate. When the rate of precipitation is equal to the rate of dissolution, equilibrium obtains and the corresponding temperatures are the equilibrium saturation temperatures (the minima in the flow trace). The unplugging mode conveniently illustrates the difference between the oscillating and the manual modes of operation.

B. Continuous or Automatic Mode of Operation

The continuous or automatic mode of operation is attributable to the French[5,6] and the British[2,6] workers. The main features of this method are that the cooling rate is controlled to maintain a certain fixed flow rate, and the temperature at the orifice is then the saturation temperature. Sodium passing through the orifice either dissolves or precipitates an infinitesimal amount of the impurities, and near equilibrium is maintained at all times. This mode of operation is especially advantageous at low saturation temperatures (<250°F).

C. Oscillating Mode of Operation

In the oscillating mode of operation,[1] the temperature of the plugging orifice is cycled through a certain range, causing the flow to cycle similarly.

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*In Fig. 2 and all other figures showing the actual temperature and flow profiles, the profiles are offset by 0.3 cm. A two-pen recorder was used to obtain these profiles. In these figures, the line drawn to interpret the temperature that corresponds to a flow maximum or a minimum is therefore offset by 0.3 cm.

The temperatures corresponding to the minima and the maxima of the flow trace are the impurity saturation temperatures. Both these temperatures should be equal; however, due to the thermal inertia of the system, there usually is a difference (see Fig. 9 later). The sodium temperature in the orifice is controlled by an on-off switch that is driven by the flow trace. A typical arrangement is shown in Fig. 3. The oscillating mode is, in fact, similar to the French-British continuous or automatic mode. The difference is in the degree of upstream precipitation. The oscillating plugging indicator cannot be operated with upstream precipitation, whereas the continuous plugging indicator can. At low concentrations of impurities, the oscillating mode automatically reverts to the continuous mode.

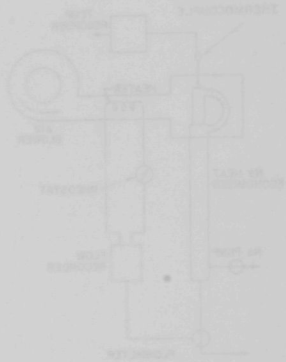

Fig. 3

Oscillating Plugging Indicator Used in Tests

III. SELECTED EXPERIMENTAL RESULTS

A. Experimental Apparatus and Procedure

The results reported in this section were obtained with a plugging-indicator circuit such as shown in Fig. 3. Figure 4 shows the orifice detail. Figure 5 shows the sodium analytical loop (SAL) used in the plugging-indicator test. The sodium in the main tank (approximately 120 lb) is maintained at 600°F. The flowmeter in the plugging loop is located between the main sodium tank and an economizer, which is not shown in Fig. 5. The economizer (see Fig. 3) is used to cool the sodium to near the impurity saturation temperature, but not lower than the saturation temperature, for the sodium would be depleted of impurities before reaching the orifice. The air blower, located

Fig. 4. Plugging-indicator Orifice Detail

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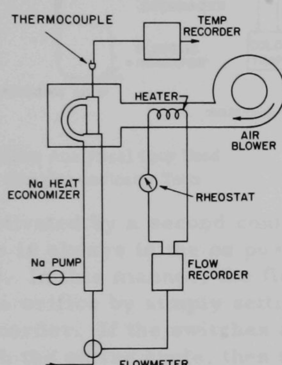

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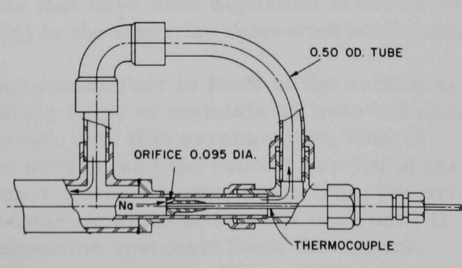

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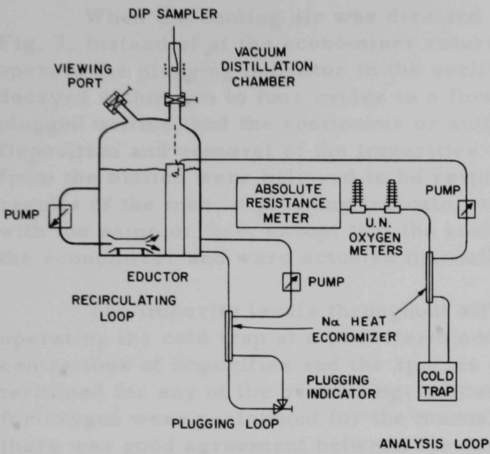

Fig. 5. Sodium Analytical Loop Used in Plugging-indicator Tests

downstream from the orifice, brings the temperature of the sodium in the orifice below the impurity saturation temperature and causes precipitation in the orifice.

When the sodium flow in the circuit decreases below a preset value, a heating element in the air blower is actuated by a control switch on the flow recorder. The heating element is maintained in the on position until some of the precipitate in the orifice goes back into solution. The sodium flow then increases to a second preset (higher) value, at which point the heating element in the air blower is deactivated by a second control switch on the same flow recorder. The air blower is always in the on position while the plugging indicator is being operated. In this manner, the flow may be cycled between any two plug fractions of the orifice by simply setting the heating element on-off switches on the flow recorder. If the switches are set so that the orifice has some deposit through the entire cycle, then the meter is said to be operating in the oscillating mode. Figures 9-13 are good examples of this operational mode. The bare-orifice flow for these traces is 0.24 gpm. If the controls are set so that the deposit is completely removed before the next cycle is executed, the meter operates in the partial oscillating or unplugging mode, as in Figs. 2 and 8. The impurity saturation temperature is the orifice temperature that corresponds to the maximum and minimum of the flow trace in the oscillatory operational mode and to the minimum of the flow trace in the unplugging mode of operation. At both the maximum and minimum of the flow trace, the rate at which impurities dissolve is equal to the rate at which they deposit. Thus, the impurities in solution are at, or very near, equilibrium with the impurities that have been deposited in the orifice, and the corresponding temperature(s) is the impurity saturation temperature.

It is not necessary to have an economizer in front of the orifice as shown in Fig. 3; the economizer merely helps to maintain an even temperature in the main sodium tank. However, with this arrangement, heat is removed at or downstream from the orifice, and the coolest portion of the loop during the cooling cycle is a short distance downstream from the orifice. Deposition starts here on the interior walls and works back until it reaches the orifice. This avoids deposition upstream from the orifice, which as noted below is disadvantageous.

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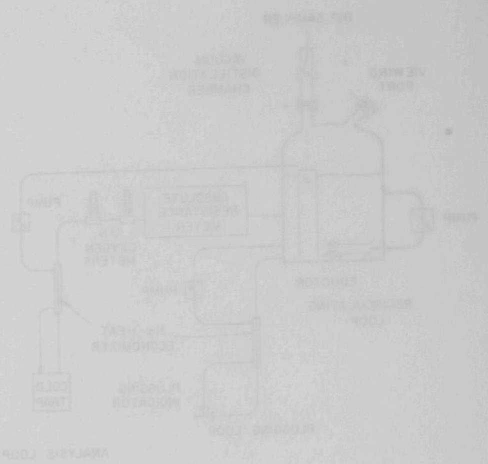

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When the cooling air was directed at the economizer, as shown in Fig. 3, instead of at the economizer return loop, it was not possible to operate the plugging indicator in the oscillating mode. The flow oscillations decayed within two to four cycles to a flow value that indicated a partially plugged orifice, and the continuous or automatic mode of operation resulted. Deposition and removal of the impurities on the economizer wall upstream from the orifice were believed to be responsible for this phenomenon. The results of the manual plugging indicator were obtained on the same system with the same orifice, except that the cooling and heating were directed at the economizer and were actuated manually.

The impurity levels throughout all our runs were controlled by operating the cold trap at a predetermined temperature. The actual concentrations of impurities and the species that were present were not determined for any of the oscillating-indicator runs. Distillation[7] analyses for oxygen were performed for the manual-indicator runs. In general, there was good agreement between the oxygen analysis whenever performed and the cold-trap temperature when an impurity was injected into the sodium system. The oxygen solubility-versus-temperature curve of Rutkauskas[3] was used.

B. Effect of Cooling Rates on Manual Plugging Temperature

Results with the SAL manual plugging indicator showed that the manual-plugging-indicator temperatures did not agree with cold-trap temperatures as required by theory. An effort was made to correlate the difference and to assess the reliability of the manual plugging indicator. In these runs, the sodium flow rate and cold-trap temperature remained nearly constant at 0.25 gpm and 456°F, respectively, and the cooling rate of sodium was varied from 0.6 to 25°F per minute. Figure 6 shows some of the results. It may be seen that the manual plugging temperature is a function of the cooling rate of sodium and poorly correlated to the cold-trap temperature. Figure 6 also shows that in runs with the lower cooling rates there is a tendency to approach the true saturation temperature. It is also true that at the lower cooling rates of sodium it is more difficult to establish where plugging started. Figure 7 shows a trace of an actual manual plugging indicator run at a cooling rate of 5°F per minute. Figure 7 also shows a difference of 50°F between plugging and unplugging (350-400°F) and a measured oxygen content of sodium of 31 ppm corresponding to a saturation temperature of approximately 460°F.

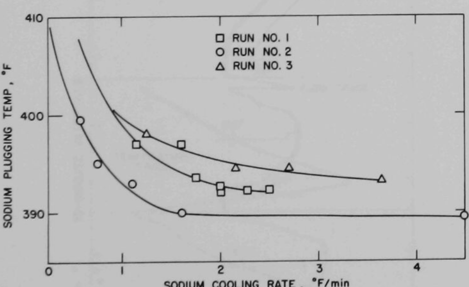

Fig. 6. Sodium Plugging Temperature vs Cooling Rate at Cold-trap Temperature of 456°F

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Results with the ANL manual plugging indicator showed that the manual-plugging-indicator temperatures did not agree with cold-trap temperatures as required by theory. An effort was made to correlate the difference and to assess the reliability of the manual plugging indicator. In these runs, the sodium flow rate and cold-trap temperature remained nearly constant at 0.25 gpm and 350°F, respectively, and the cooling rate of sodium was varied from 0.6 to 23°F per minute. Figure 6 shows some of the results. It may be seen that the manual plugging temperature is a function of the cooling rate of sodium and poorly correlated to the cold-trap temperature. Figure 6 also shows that in runs with the lower cooling rates there is a tendency to approach the true saturation temperature. It is also true that at the lower cooling rates of sodium it is more difficult to establish where plugging started. Figure 7 shows a trace of an actual manual plugging indicator run at a cooling rate of 8°F per minute. Figure 7 also shows a difference of 50°F between plugging and unplugging (350-400°F) and a measured oxygen content of sodium of 51 ppm corresponding to a saturation temperature of approximately 460°F.

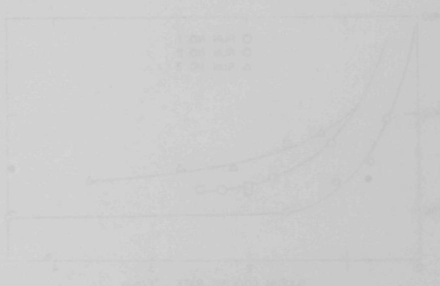

Fig. 6. Sodium Plugging Temperature vs Cooling Rate at Cold-trap Temperature of 350°F

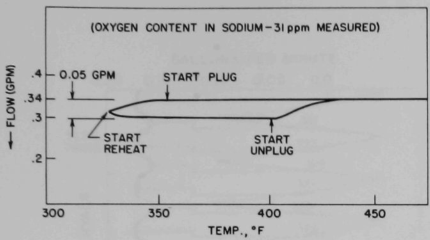

Fig. 7. Plot of Actual Manual-plugging-indicator Run

In summary, the manual plugging indicator is a poor device for determining sodium saturation temperatures.

C. Results with the Oscillating-mode Plugging Indicator

Figures 8-17 are reproduced segments of the actual recorded temperature and flow profiles. Figures 2 and 8 show some unplugging runs made to compare the plugging temperature obtained with a manual plugging indicator with that of the oscillating indicator. The reason for the difference has already been explained by others[1] and was discussed briefly in Section III.

Figures 9-13 show the temperature and flow traces for a cold-trapping run. Starting with the sodium impurity-saturation temperature at 360 ± 10°F, the cold trap was operated to reduce the impurities to the lowest limit possible. The oscillating plugging indicator was turned on and operated continuously (automatically) through the entire run, a period of some 18 hr. Figure 9 shows the plugging-indicator trace at the beginning of this run, and Figs. 10-13 were taken in that order from the same trace

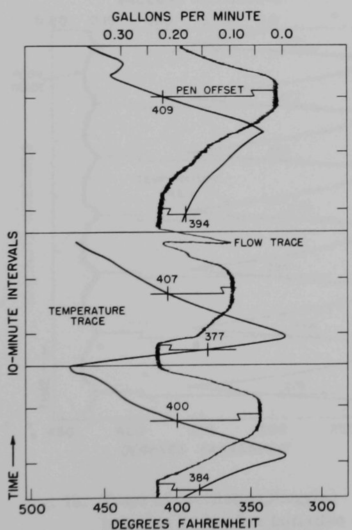

Fig. 8. Effect of Cooling Rate on the Interpretation of Manual-plugging-indicator Temperature

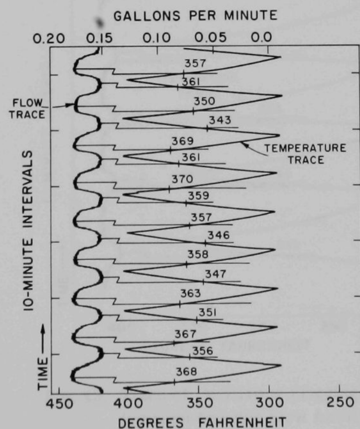

Fig. 9. Trace of Oscillating Plugging Indicator at Start of a Cold-trapping Run

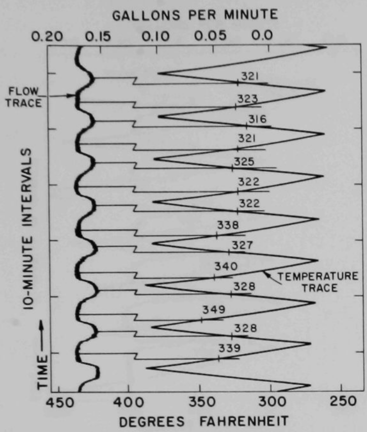

Fig. 10. Trace of Oscillating Plugging Indicator 2 hr after Initiation of Cold Trapping

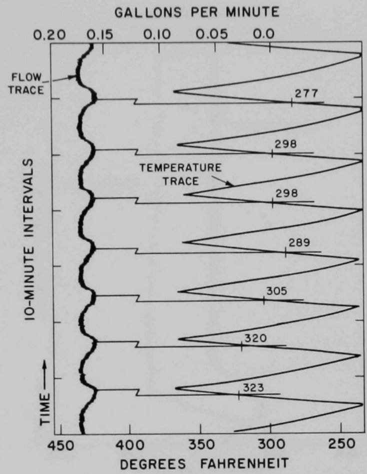

Fig. 11. Trace of Oscillating Plugging Indicator 4 hr after Initiation of Cold Trapping

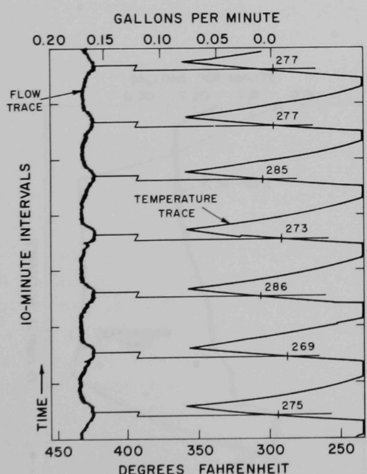

Fig. 12. Trace of Oscillating Plugging Indicator 14 hr after Initiation of Cold Trapping

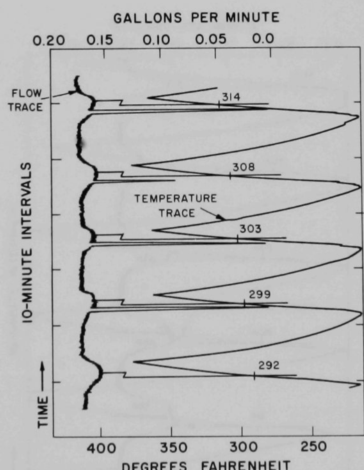

Fig. 13. Trace of Oscillating Plugging Indicator Showing Flow Arrest due to Sodium Freezing

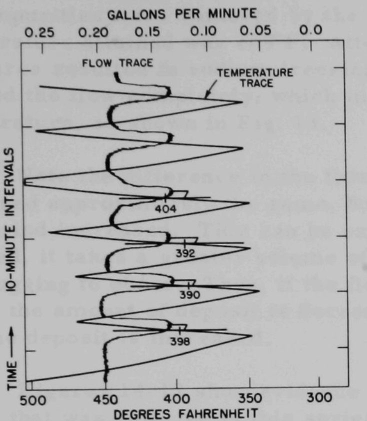

Fig. 14. Trace of Oscillating Plugging Indicator, Showing Evidence for a Two-species Precipitation Process

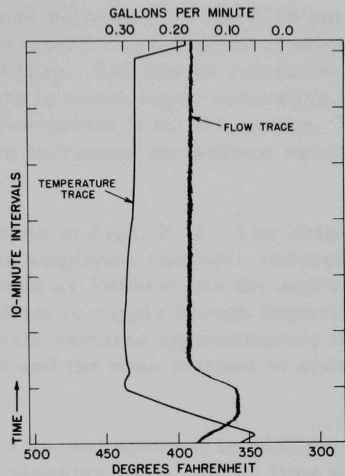

Fig. 15. Trace of Oscillating Plugging Indicator, Arrested at Approximately 440°F to Prove Two-species Precipitation Phenomenon

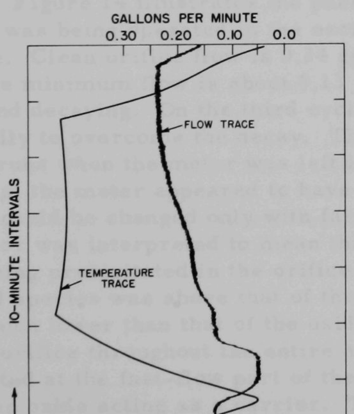

Fig. 16. Trace of Oscillating Plugging Indicator, Arrested at Approximately 480°F to Prove Two-species Precipitation Phenomenon

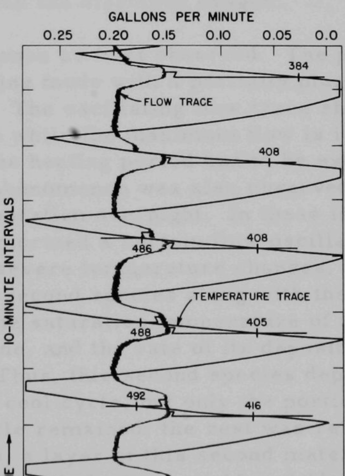

Fig. 17. Trace of Oscillating Plugging Indicator Interpreted for Two Impurity Species

at later intervals. The flow trace remained between 0.15 and 0.18 gpm throughout the entire run, while the temperature trace dropped, indicating that impurities were removed by the cold trap. The lowest saturation temperature attained was 269°F. Attempts to reach lower saturation temperatures resulted in sodium freezing downstream from the orifice. This blocked the flow completely, which in turn increased the sodium saturation temperature, as shown in Fig. 13.

Note the difference in the flow traces in Figs. 9-12. The shape has remained approximately the same, but the amplitude has been reduced and the period increased. This can be explained as follows: As the sodium is purified, it takes a greater volume of sodium to supply enough impurities for plugging to occur. Thus, if the flow rate remains approximately the same, the amount of deposit is decreased and the time it takes to accumulate the deposit is increased.

Figures 14-17 show evidence for a second species, in addition to the oxide, that was detected in this series of plugging runs. At the time of this observation, the sodium saturation temperature was approximately 450°F. The plugs were never removed for examination and identification of the second species or of the oxide. This second species is characterized by being much slower to precipitate and redissolve than the oxide, and by blocking a smaller portion of the flow. Thus, this species probably is present in smaller amounts in sodium than the dissolved oxygen.

Figure 14 illustrates the phenomenon as first observed. The plugging meter was being operated in the oscillating mode with a partially plugged orifice. Clean orifice flow is 0.24 gpm. The oscillating flow trace shows that the minimum flow is about 0.13 gpm while the maximum flow is irregular and decaying. On the third cycle, the heating period had to be extended manually to overcome the decay. This phenomenon was also observed in other runs when the meter was left in operation overnight. In these instances, the meter appeared to have memorized a certain flow oscillation, which could be changed only with fairly severe temperature changes. This behavior was interpreted to mean that a second species along with the oxide was being precipitated in the orifice. The saturation temperature of the second species was above that of the oxide, and the rate of its deposition was much lower than that of the oxide. Thus, this second species deposited in the orifice throughout the entire heat-cool cycle, but only the portion deposited at the fast-flow part of the cycle remained; the rest was removed, with the oxide acting as a carrier. Thus, a layer of this second material was built up, covering the oxide not being cycled. Figures 15 and 16 illustrate this phenomenon. Figure 15 shows a flow trace following several hours of plugging-indicator operation, after which the automatic cycling was suppressed and the temperature at the orifice was maintained at approximately 440°F for one hour. The flow returned to the high rate of the cyclic operation and remained fairly constant. Whenever this is done with cleaner sodium, the flow returns to 0.24 gpm (unplugged-orifice condition). In the at later intervals. The flow trace remained between 0.15 and 0.18 gpm throughout the entire run, while the temperature trace dropped, indicating that impurities were removed by the cold trap. The lowest saturation temperature attained was 267°F. Attempts to reach lower saturation temperatures resulted in sodium freezing downstream from the orifice. This blocked the flow completely, which in turn increased the sodium saturation temperature, as shown in Fig. 13.

Note the difference in the flow traces in Figs. 9-12. The shape has remained approximately the same, but the amplitude has been reduced and the period increased. This can be explained as follows. As the sodium is purified, it takes a greater volume of sodium to supply enough impurities for plugging to occur. Thus, if the flow rate remains approximately the same, the amount of deposit is decreased and the time it takes to accumulate the deposit is increased.

Figures 14-17 show evidence for a second species, in addition to the oxide, that was detected in this series of plugging runs. At the time of this observation, the sodium saturation temperature was approximately 450°F. The plugs were never removed for examination and identification of the second species or of the oxide. This second species is characterized by being much slower to precipitate and redissolve than the oxide, and by blocking a smaller portion of the flow. Thus, this species probably is present in smaller amounts in sodium than the dissolved oxygen.

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Figure 17 shows a flow trace with what appears to be two species, oxide plus the second species, being deposited and dissolved in appreciable amounts so that the history of one substance is superimposed on the other.

IV. CONCLUSIONS AND RECOMMENDATIONS

The manual plugging indicator is a poor device and the oscillating and the continuous plugging indicators are very useful devices for measuring impurity saturation temperatures in sodium circuits.

The oscillating and continuous plugging indicators can be used simply for monitoring purposes, or they can also be used for studies of impurity behavior in sodium with respect to cold trapping. A removable-orifice plugging indicator would be very helpful, and efforts should be extended in this direction. Also, studies on precipitation rates of the different species and on seeding of orifices could extend the range of usefulness of plugging indicators.

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We wish to express our appreciation to John T. Holmes and Milton Ader for their valuable suggestions and ideas in preparing this report, and to L. G. Bartholme and R. D. Baldwin for their assistance in constructing and operating the Sodium Analytical Loop.

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